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3-77-61-2

# THE EFFECTS OF HIGH ENERGY RADIATION ON INFRARED OPTICAL MATERIALS

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3-77-61-2

## THE EFFECTS OF HIGH ENERGY RADIATION ON INFRARED OPTICAL MATERIALS

#### AN ANNOTATED BIBLIOGRAPHY

Compiled by
GEORGE R. EVANS
and
WILLIAM E. PRICE

SPECIAL BIBLIOGRAPHY SB-61-25

**MAY 1961** 

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#### ABSTRACT

A compilation of abstracts on the effects of high energy radiation on the infrared and other optical properties of germanium, silicon, silica, glass and quartz. Pertinent periperal matter is included.

#### SOURCES CHECKED

In addition to using the facilities of the Technical Information Center, Lockheed Missiles & Space Division and ASTIA (Armed Services Technical Information Agency), the following index were checked.

INSTITUTE OF METALS. JOURNAL

NUCLEAR SCIENCE ABSTRACTS

RADIATION EFFECTS INFORMATION CENTER. ACCESSION LIST

SCIENCE ABSTRACTS. SECTION A: PHYSICS

SEMICONDUCTOR ELECTRONICS

SEMICONDUCTORS AND PHOSPHORS

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LOCKHEED AIRCRAFT CORPORATION

#### INTRODUCTION

The infrared transmission of optical materials has been the subject of very few investigations. Ordinarily the absorption spectra is of most interest because it reveals energy levels of defects and bonding types of solid materials. The following compilation of abstracts is an attempt to gather in one report most of the references on the effects of high energy radiation on the infrared optical properties of specific materials. The materials are silicon, germanium, silica, glass and quartz. The effort was concentrated on finding changes in transmission properties but many periperal subjects are included. The period covered is from 1950 to the present date.

The abstracts are arranged by author under each specific material. A corporate author index is offered as a help in following a set of progress reports for a single company.

#### GERMANIUM

Huldt, L. and Staflin, T.
Optical constants of evaporated films of zinc sulphide and germanium in the infrared. OPTICA ACTA v. 6, p. 27-36, Jan 1959.

Measurements of the optical constants of vacuum-evaporated films of zinc sulphide and germanium some microns thick by three different methods in the region  $2000 - 10,000 \, \mathrm{cm}^{-1}$  (5 - 1 $\mu$ ) are reported. The refractive indices of both substances proved to be definitely higher than for the corresponding bulk material. When the films were evaporated with pure nitrogen as residual gas instead of air, the refractive index decreased considerably and reached, for ZnS, approximately the bulk value, whereas for Ge it still remained somewhat higher. The variations in the modes of determination seem to exclude the observed anomalies being due to the complexity of the film structure.

2. Meyer, H. J. G.

Theory of infrared absorption by conduction
electrons in germanium. JOURNAL OF THE PHYSICS

AND CHEMISTRY OF SOLIDS v. 8, p. 264-269, Jan 1959.

It is shown that a theory of infrared absorption by conduction electrons which takes into account the structure of the conduction band and acoustical as well as optical intravalley scattering can be developed without any serious approximation. In combination with an estimate of the possible influence of impurity scattering, the theoretical results can be used for the determination of one of the acoustical and of the optical deformation potential constants. From available experimental data the approximate numerical value of the latter is determined. The general limits of validity of the theory are discussed.

3. Nazarewicz, B. W.

INDIRECT ABSORPTION EDGE IN GERMANIUM BOMBARDED BY FAST NEUTHONS. Polish Academy of Sciences,

#### Institute of Nuclear Research,

NP-8661, 1959.

This paper reports the results of absorption measurements for germanium single crystals with various concentrations of structural defects introduced by fast neutron bombardment. It was found that the edge is of the form expected for indirect transitions accompanied by phonon interaction.

An analysis of absorption data on the basis of the Macfarlane - Roberts formula yield values for the indirect energy gap and the temperature of the phonon involved.

Various concentrations of defects were obtained by partially annealing at various elevated temperatures.

4. Philipp, H. R. and Taft, E. A.

Optical constants of germanium in the region

1 to 10 EV. PHYS. REV. v. 113, p. 1002-1005,

15 Feb 1959.

The reflectance,  $|\mathbf{r}(\lambda)|^2$ , of single-crystal germanium has been measured in the range 0.6 to 11.3ev and the phase,  $\theta(\lambda)$ , has been computed from these data using the Kramers - Kronig relation between the real and imaginary parts of the complex function In  $\mathbf{r} = \mathbf{In} |\mathbf{r}| + \mathrm{i}\theta$ . The optical constants, n and k, have then been determined from the Fresnel reflectivity equation. The real part of the refractive index, n, has maxima of 5.5 at 2.07 ev and 4.2 at 3.2 ev. Above 6 ev, the index is very nearly 1. The extinction coefficient, k, shows maxima of 2.0 at 2.5 ev and 4.2 at 4.4 ev. Below 3.5 ev, the results are in good agreement with measurements of Archer (see 2017). Beyond this point, they depart from his extrapolated curve.

5. Purdue Research Foundation

SEMICONDUCTOR RESEARCH. QR no. 1, 1 July-

30 Sep 54. (Contract DA-36-039-sc 63222).

ASTIA AD-54 914.

Optical Absorption in N-type Germanium: This study was made in the wavelength from 5-30 microns. The samples of different carrier concentrations were at three temperatures:  $450^{\circ}\text{K}$ ,  $294^{\circ}\text{K}$  and  $78^{\circ}\text{K}$ .

A comparison of the room temperature and liquid nitrogen temperature absorption curves for a given sample show that the absorption coefficient at 78°K is even higher than at 294°K for sufficiently small wave numbers.

At 450°K if the scattering is predominately lattice scattering then theory predicts that "a" is proportional to "n" and the absorption should have a slope of -2.3. The correspondence between theory and experiment indicates an effective electron mass m\*=.13m where m is the free electron mass.

6. Stoeckmann, F., et al.

Photoconductivity of electron bombarded Ge

(Abstract of meeting). PHYSICAL REVIEW

v. 98, n. 5, p. 1535, 1 June 1955.

The photoconductivity of n-type Ge illuminated with light in the fundamental absorption region, increases during bombardment with fast electrons (6 MeV) at 90 K. The increase is fast at the start of irradiation, slower after prolonged bombardment amounting to two orders of magnitude. The time constants of the rise and decay curves are about one second. The photoconductivity decreases as the sample is converted to p-type. These observations show the production of hole traps by the bombardment.

7. Whanov, Y. I.

An investigation of infrared absorption

by minority carriers in germanium. SOVIET

PHYS. - SOLID STATE v. 1, p. 326-329, Mar 1959.

An investigation of the absorption spectra due to hole injection in germanium over the wavelength region from 2 to 13µ at room temperature and at T = 105°K is reported. Two weak maxima at 3.4µ were observed at room temperature. At wavelengths above 5.5µ the absorption increased sharply, remaining constant over the region 9 to 13µ. At T=105°, instead of two maxima at 3.4 µ and 4.7µ, a narrow absorption band was observed at 4µ with absorption comparable to that observed at room temperature. On either side of the maximum the absorption decreased to vanishingly small values. In the region from 6µ the absorption increased smoothly, reaching a maximum at 12µ approximately equal to the room temperature value. There is good agreement between the absorption spectra due to injection carriers in n-type germanium and the absorption spectra due to equilibrium carriers in p-type germanium.

#### STLICON

8. Becker, M., Fan, H. Y. and Lark-Horovitz, K.

Infrared absorption of nucleon bombarded

silicon I (Abstract of Meeting) BULLETIN

OF THE AMERICAN PHYSICAL SOCIETY v. 26,

n. 6, p. 43, 24 Oct 1951.

Recent investigations show that the absorption in silicon, beyond 1.1 micron, rises again toward longer wave lengths and is smaller the higher the resistivity.

9. Cole, R. L. and Mitchell, G. A.

DEVELOPMENT OF SILICON INFRARED OPTICAL

COMPONENTS. Texas Instruments, Inc., Dallas.

Quarterly progress rept. no. 4, 1 Sep-1 Dec 59.

(Contract AF 33(600)380085). ASTIA AD-234 420.

Descriptors: Infrared optical materials\*; Silicon\*; Casting; Infrared domes\*; Infrared windows; Radiation effects; Molding materials; Neutron bombardment; Silicon compounds\*; Monoxides; Nitrides.

This Document includes:

Romanko, J.

EFFECTS OF REACTOR RADIATION ON THE OPTICAL PROPERTIES OF SILICON AND SILICON MONOXIDE.

Convair, Fort Worth, Tex. Rept. no. FZK-118.

(Contract AF 33(600)38085).

A casting process has been developed to produce silicon domes and plates up to 4 in. in diameter. The same process will be used to cast 12 in. -diam

domes and plates. Work continues on the development of structurally sound, pure silicon mitride molds. These molds would be used in conjunction with the malt or pour casting method of obtaining silicon ingots. Design and fabrication nears completion on the hot forming machine which will be used to thermally deform plates of silicon. An epoxide easting resin still appears to be the most suitable adhesive for joining or bonding silicon to itself. After irradiation, joined silicon samples were found to increase in bond strength some 30 to 40%. Rept. no. FZK-118: Specific matters of interest in this effort, which emphasizes the 2-to 6-u region, include: (1) a study of the literature on the effects of nuclear radiation on silicon optics: (2) the high-temperature characteristics of very high-resistivity silicon, such as might be obtained by neutron bombardment; (3) the expected radiation environment to which such equipment could be subjected and expected to function; and (4) the use of neutron bombardment to raise economically the electrical resistivity of low-grade silicon to a level suitable for optical use.

10.

Cole, R. L.

DEVELOPMENT OF SILICON INFRARED OPTICAL

COMPONENTS. Texas Instruments, Inc., Dallas.

Quarterly progress rept. no. 6, 1 Mar-1 June 60, 38p.

(Contract AF 33(600)38085). ASTIA AD-240 268.

Descriptors: Infrared optical materials\*; Silicon\*; Infrared domes\*; Casting; Infrared windows; Radiation effects; Molding materials; Silicon compounds\*; Monoxides; Nitrides; Vapor plating; Hydraulic presses.

A casting process has been developed which will consistently produce 12 inch diameter x 5/8 inch thick cast silicon plates, and  $8\frac{1}{2}$  inch outside diameter  $x \stackrel{1}{\downarrow}$  inch thick cast silicon domes. A large hydraulic press and furnace combination has been completed. This machine was designed specifically to thermally deform cast silicon plates into spherical segments. The segments are then bonded into  $7\frac{1}{2}$  inch outside diameter x  $\frac{1}{4}$  inch thick mosaic silicon domes. In order to supply silicon plates and segments for the large mosaic domes and plates, the cast plates were all produced on a small casting machine previously built and normally used for producing four-inch diameter domes. The silicon mosaic structures will receive a final process which involves grinding, polishing and coating. Two four-inch diameter silicon vapor deposited domes have been produced, but have slightly low transmission. They are mechanically strong with no visible voids or inclusions. In addition, an appreciable reduction in adsorption at nine microns was realized. Strength tests performed on pressed silicon samples show virtually no change in the moduli of rupture and elasticity over values obtained with cast or grown silicon samples.

11.

Cole, R. L., Mitchell, G. and Ricks, J.

DEVELOPMENT OF SILICON INFRARED OPTICAL

COMPONENTS (TRANSMITTING WINDOWS) Texas

Instruments, Inc., Dallas. Final technical

engineering rept. AMC TR 60-7-719. 1 Nov

58-25 Aug 60. Dec 60, 211p. (Contract

AF 33(600)38085, Proj. 7-719). ASTIA

AD-250 513.

Descriptors: \*Infrared windows, \*Infrared optical materials, \*Silicon, Casting, Mechanical properties, Production, Radiation effects, \*Semiconductors, Impurities, Silicon compounds, Monoxides.

This rept. Includes:

Romanko, J.

EFFECTS OF REACTOR RADIATION ON THE OPTICAL PROPERTIES OF SILICON AND SILICON MONOXIDE.

Convair, Fort Worth Tex. Dec 60, 21p.

(Appendix A to AMC TR 60-7-719).

Hill, O. H. .

EXPERIMENTAL STUDIES OF THE EFFECTS OF REACTOR RADIATION ON THE UP GRADING OF IMPURE SILICON. Convair, Fort Worth, Tex. Dec 60, 26p. (Appendix B to AMC TR 60-7-719).

Silicon plates up to 12 inches in diameter by 1 inch thick and silicon hemispherical domes up to  $8-\frac{1}{2}$  inches in diameter by  $\frac{1}{4}$  inch thick can be economically cast or segmented for use as infrared transmitting windows. The optical transmission of nuclear reactor irradiated silicon showed some improvement for limited wavelengths.

12

Corbett, J. W.

DEFECTS IN IHRADIATED SILICON II. TNFRARED ABSORPTION OF THE S1-A CENTER. General Electric Research Laboratory, Schenectady, New York. 60-R-(2559 M) Oct 60, 27p.

Also In THE PHYSICAL REVIEW v. 121, n. 4, p. 1015-1022, 15 Feb 1961.

The Si-A center is a major, radiation-damage defect produced in "pulled" silicon by room temperature irradiation. In this report (II), we present the infrared measurements which, in conjunction with the spin resonance measurements of the preceding report (i), establish the identity of the Si-A center. A new infrared absorption band is observed at 12 u in electronirradiated silicon. This band is shown to be a vibrational band of impurity oxygen in the lattice. Macroscopic and microscopic correlations between the 12 µ band and the spin resonance of the Si-A center are presented. The macroscopic correlations are of production rate, recovery etc. The microscopic correlations derive from the absorption of polarized infrared radiation by samples of various crystallographic orientations, subjected to a uniaxial. compressive stress. Partial alignment of the defects is induced by the stress and is detected as a dichroism in the 12 µ band. This alignment is compared to the corresponding alignment studies in the spin resonance measurements in report I. It is shown that the kinetics and magnitude of the response to the stress are the same for the defects observed in both types of measurements. This shows that the 12 µ band arises from the Si-A center and established the configuration of the oxygen in the defect. These results, together with the results of report I, allow us to conclude that the Si-A center is a lattice vacancy with an oxygen atom bridging two of the four broken bonds associated with the vacancy. The remaining two bonds can trap an electron giving rise to the spin resonance spectrum of the defect. The identification of the Si-A center indicates that the vacancy is mobile in a room-temperature irradiation.

Corbett, J. W., Watkins, G. D. and Chrenko, R. M.
Infrared studies of electron irradiated
silicon I. BUIL. AM. PHYS. SOC. v. 5, Ser. II,
p. 25-26 (A), 27 Jan 1960.

The IR absorption spectrum  $(1.4-14\mu)$  of silicon irradiated at room temperature with 1.5 mev electrons was discussed. A sharp band at 12  $\mu$  with an intensity

roughly proportional to the concentration of dissolved oxygen originally in the silicon has been observed. The production rate of this band vs. irradiation correlates with that of the defect previously studied in spin resonance (A center) and in electrical measurements (net acceptor at  $E_{\rm c}$ =0.17 eV), which has been shown to be strongly dependent upon exygen content. The annealing behavior or the 12  $\mu$  band is similar to that of the other measurements, again suggesting that they arise from the same defect. In a sample enriched with 14 percent 0<sup>10</sup>, a small subsidiary peak is observed which identifies the 12  $\mu$  absorption as arising from the vibration of a single exygen atom. These data lend support to the model proposed from spin resonance studies that the A center, which is the dominant defect produced by irradiation in pulled silicon, involves an exygen atom trapped in a lattice vacancy.

14. Fan. H. Y.

Infrared absorption of nucleon bombarded silicon II (Abstract of meeting). BULLETIN OF THE AMERICAN PHYSICAL SOCIETY v. 26, n. 6, p. 34, 24 Oct 1951.

The reduction of long wavelengths absorption to very small values is convincing evidence for ascribing the long wavelength absorption of free carriers.

15. Fan. H. Y.

REPORT. Purdue University, Purdue Research Foundation, Lafayette, Indiana. PRF-1258, Progress Report, 14th Quarterly Report.

1 Nov 59-31 Jan 60. ASTIA AD-235 332.

Several additional lattice absorption bands were observed in electron or neutron bombarded silicon between 9-14 microns. A summary is given of the investigation of electron spin resonance in neutron irradiation silicon.

16.

Fan, H. Y. and Ramdas, A. K.
Infrared absorption in neutron-irradiated
silicon. J. PHYS. CHEM. SQLIDS v. 8,
p. 272-274. Jan 1959.

Irradiation by high energy neutrons and electrons produces in silicon infrared absorption bands associated with the introduced lattice defects. Several bands have been observed. The possibility of observing a particular absorption band depends on the Fermi level in the irradiated sample which determines the state of ionization of the defects. The absorption was studied after annealing the sample at different temperatures. It was found that the various absorption bands are associated with different types of defect centers. One of the bands, at 20.5 microns, increased or decreased with temperature annealing depending on the Fermi level. The observation is understandable on the assumption that the anneal of the type of absorption center depends upon the state of ionization of the defects.

Hill, O. H., Cole, R. L. and Mitchell, G. A.

DEVELOPMENT OF SILICON INFRARED OPTICAL

COMPONENTS. Texas Instruments, Inc., Dallas.

Quarterly progress rept. no. 5, 1 Dec 59
1 Mar 60, 83p. (Contract AF 33(600)38085).

ASTIA AD-237 392.

Descriptors: Infrared optical materials\*; Silicon\*; Infrared domes\*; Casting; Infrared windows; Padiation effects; Molding materials; Silicon compounds\*; Monoxides; Nitrides.

A casting process was developed to produce silicon domes and plates up to 6 in. in diameter. This same process will be used to cast  $8\frac{1}{2}$ -in. -diam silicon domes and up to 12-in. diam silicon plates. The fabrication of the hot forming machine nears completion. This machine will be used to press 43/4-in. -diam by 7/16-in. -thick cast silicon plates into spherical segments which will be used to form  $7-\frac{1}{2}$ -in. - diam mosaic silicon domes. Cast silicon plates, 43/4-in. in diam by various thicknesses, will be used to form the required 12-in. -diam mosaic plates. The effect of irradiation on silicon monoxide coatings and the effect of irradiation on the absorption coefficient of low resistivity silicon was investigated. Irradiation of silicon monoxide coatings showed little difference before and after irradiation. However, the effect of irradiation on the absorption coefficient of low

resistivity silicon was pronounced. Low resistivity natype and p-type silicon showed a transformation after irradiation that markedly reduces their absorption coefficient. The reduction was non-linear with a small reduction at  $2\mu$  to a large reduction at  $4-\frac{1}{2}$  to  $6\mu$ .

18.

Longo, T. A.

NUCLEON IRRADIATION OF SILICON SEMICONDUCTORS.

A thesis Submitted to the Faculty of Purdue

University. Aug 1957.

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Changes in Mobility of Charge Carriers Produced by Deuteron Irradiation of Silicon

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19. Longo, T. A. and Kleitman, D.

Neutron irradiated silicon (abstract of

meetings) PHYSICAL REVIEW v. 100, n. 4,

p. 1260-1261, 15 Nov 1955.

After fast neutron irradiation at 45 C the temperature dependence of Hall coefficient and resistivity of both n-type and p-type single crystal silicon samples indicated that intrinsic behavior was approached in each sample. A 1.75  $\mu$  peak in the optical absorption coefficient was observed. Appreciable photoconductivity was observed beyond the fundamental region to about 1.5  $\mu$  after which there is a sharp drop with a shoulder in the region between 2 and 3  $\mu$ . Annealing experiments were continued.

20. Purdue University. Physics Dept.

SEMICONDUCTOR RESEARCH. Second Quarterly

Report. 1 Oct-1 Dec 54. (DA 36-939-sc63222).

Effects of Annealing in Neutron Irradiated Silicon. Infrared optical absorption measurements were made on p-type neutron bombarded sample of Silicon II after certain amounts of annealing. After annealing the 1.8µ absorption peak decreases and appears to acquire a long wavelength tail, both at room temperature and at L. N.

21. Purdue Research Foundation.

SEMICONDUCTOR RESEARCH. QR no. 12, 1 May-

31 July 59. (DA 36-039-sc 71131). ASTIA

AD-230 803.

Infrared absorption in irradiated silicon, by A. K. Ramdas and H. Y. Fan.

22. • Vavilov, V. S.

Absorption of infrared radiation by free charge carriers in silicon. FIZ. TVERDOGO TELA v. 2, n. 2, p. 374-7, Feb 1960.
(In Russian)

Reports an investigation (at  $295^{\circ}$ K) of the absorption of 1-11  $\mu$  radiation in silicon monocrystals of p- and n-type with free carrier densities from  $10^{14}$  to  $5 \times 10^{17}$  cm<sup>-3</sup> and in silicon previously irradiated with fast neutrons (carrier density  $10^{12}$  cm<sup>-3</sup>). Comparison with Fan's theory (Abstr. 8771 of 1956) gave a mean effective hole mass of  $3.6 \times 10^{-28}$ g. It was found that Fan's relationship  $\alpha \sim \lambda 3/2$  ( $\alpha$  is the absorption coefficient and  $\lambda$  is the wavelength of infrared radiation) was not satisfied in n-type crystals and in highly transparent p-type crystals.

Vavilov, V. S., Plotnikov, A. F. and Zakhvatin, G. V. Infra-red absorption of high-resistivity silicon with radiation-induced defects. FIZIKA

TVERDOGO TELA v. 1, n. 6, p. 976-978, 1959.

(In Russian). SOVIET PHYSICS-SOLID STATE

v. 1, n. 6, p. 894-895, 1959.

The absorption coeff.,  $\alpha$ , in the  $\lambda$  range 1-10 $\mu$ , were determined for p-type Si before and after irradiation with fast (~1 MeV) neutrons, the electresistivity of the defect-free and irradiated specimens being 65 and 2 x 105  $\Lambda$ .cm, resp. In the 2·2-7- $\mu$  range,  $\alpha$  of the irradiated specimens at room temp. was  $\alpha$  0.1/cm. In addn. to the absorption zone (with a max. at 1·8  $\mu$  at room temp. and at 1·7  $\mu$  at 20°K) due to lattice vibrations, another absorption zone (with a max. at 9  $\mu$ ), associated with the presence of 0, was observed.

Watkins, G. D., Corbett, J. W. and McDonald, R. S.

Infrared studies of electron irradiated silicon

II. BULL. AM. PHYS. SOC. v. 5, Ser. II,

p. 26(A), 27 Jan 1960.

The annealing of the 12µ band produced in silicon by room temperature irradiation with 1.5 mev electrons (see 6050) was discussed. The first stage

( $\sim 300^{\circ}$ C) consists of the disappearance of the 12 $\mu$  band and the emergence of a new band at 11.3 $\mu$  of approximately the same intensity. It is primarily in this stage that the spin resonance center anneals and electrical properties recover. It is clear, however, that the sample has not been returned to its initial state. During subsequent higher temperature annealing ( $\sim 500^{\circ}$ C), the 11.3 $\mu$  band disappears and a succession of bands between 11.3 and 9 $\mu$  band emerge and disappear. During the second stage the 9 $\mu$  band associated with oxygen in its original state is also observed to diminish. A model of these annealing stages which is also consistent with the identification of the 12 $\mu$  band as arising from an oxygen trapped in a lattice vacancy was presented.

Watkins, G. D. and Corbett, J. W.

DEFECTS IN IRRADIATED SILICON I. ELECTRON

SPIN RESONANCE OF THE Si-A CENTER. General

Electric Research Laboratory, Electron Physics

Research; Metallurgy and Ceramics Research,

Schenectady, New York. Report no. 60-RL
(2558E), Oct 60, 48p. Also In PHYSICAL

REVIEW v. 121, n. 4, p. 1001-1014, 15 Feb 1961.

The Si-A center is a major, radiation-damage defect produced in "pulled" silicon by a room temperature irradiation. As a result of studies described in this report (I), and the following one (II), it is concluded that this center is a lattice vacancy with an caygen atom impurity bridging two of the four broken bonds associated with the vacancy. Spin resonance and electrical activity arise from an electron trapped in the other two bonds. In this report (1), the spin resonance studies are described. A molecular orbital treatment of the trapped electron wave-function satisfactorily accounts for the observed g-tensor, as well as the hyperfine interaction observed with neighboring 4.7 per cent abundant Si<sup>29</sup> nuclei. Study of the changes in the spectrum of a sample subjected to uniaxial stress are also described. Under stress, the amplitudes of the individual resonance components (which correspond to different orientations of the defect in the crystal) are observed to change. This results from (1) electronic redistribution of the trapped electrons among the defects, and thermally activated reorientation of the defects themselves under the applied stress. These two effects are separated and a quantitative study of their magnitudes and signs, as well as their rates, is given. The results confirm many of the important microscopic features of the model.

#### GERMANIUM AND SILICON

Arizona State University, Tempe, Arizona

RADIATION EFFECTS IN SOLIDS - AN INVESTIGATION

OF THE ELECTRICAL AND OPTICAL PROPERTIES OF

SEMICONDUCTORS AND INSULATORS WHICH HAVE BEEN

IRRADIATED BY FAST NEUTRONS. Final Report AT

(11-1)715. Oct 60.

This study indicates that TiO<sub>2</sub> can receive a rather large exposure to fast bombardment with Little effect on electrical conductivity. The results of this work indicate that an exposure of approximately 10<sup>17</sup> fast neutrons/cm<sup>3</sup> are necessary to produce a noticeable effect in the resistivity of the reduced samples.

27. Boltax, A.

Behavior of semiconductor and magnetic
materials in radiation environment. ELECTRICAL
MANUFACTURING. Mar 1959, p. 90-95.

Theoretical and experimental behavior of several semiconductor and magnetic materials under irradiation. Charts and graphs are presented to illustrate results obtained under specific conditions. Techniques for minimizing radiation effects are also presented.

28. Crawford, J. H., Jr.

International union of pure and applied solid state physics in electronics and telecommunications. PROCEEDINGS OF THE CONFERENCE, PRUSSELS, 2-7 June 1958.

ACADEMIC PRESS v. 4: Magnetic and optical properties, pt. 2, p. 797-807, 1960.

The influence of radiation-induced defects on the behaviour of diamondstructured semiconductors and devices.

Fan, H. Y., et al

SEMICONDUCTOR RESEARCH. Purdue Research

Foundation, Lafayette, Ind. Quarterly

rept. no. 15. Rept. no. PRF 1258. 1 Feb
30 Apr 60, 43p. (Contract DA 36-039-sc71131). ASTIA AD-239 759.

ELECTRICAL AND OPTICAL PROPERTIES: Investigation of lattice scattering mechanisms in p-type germanium from hot carrier studies. The dependence of mobility of hot carriers on carrier concentration and crystal orientation, Electric field induced hot carrier modulation of infrared radiation, Effect of lithium diffusion on impurity conduction in silicon, Microwave conductivity of semiconductors, and Analysis of resistivity in single crystal tellurium. IRRADIATION EFFECTS: Oxygen dependent absorption bands on irradiated silicon, Spin resonance in neutron irradiated silicon, and Paramagentic resonance investigation in irradiated silicon. LOW TEMPERATURE STUDIES: Adiabatic demagnetization cryostat for low temperature resistivity measurements, Magnetic susceptibility, and Transport properties of germanium at low temperatures. GENERAL: Choice of gauge in perturbation theory, Perfection of silicon crystals, Silicon single crystals, and Dislocation free germanium crystals.

Fan, H. Y. and lark Horovitz, K.

IRRADIATION OF SEMICONDUCTORS. Purdue

University Department of Physics.

27 Aug-7 Sep 56. (Contract DA 36-039-sc-71131, AT(11-1)-125).

Germanium semiconductors of various resistivities and carrier concentrations have been irradiated with 9.6 Mev deuterons 4.5 Mev polonium d-particles and

4.5 Mev electrons. The energy levels introduced by defects have been determined from the removal rate of carriers as a function of the Fermi level in the semiconductors, from the temperature dependence of Hall coefficient and resistivity and from the spectral response of photoconductivity. All this information leads to a model of the defects, in general agreement with the conclusion that interstitials act as donors, vacancies act as acceptors, and that each interstitial and each vacancy can introduce several levels.

The irradiation of silicon with neutrons, as well as with electrons and deuterons indicates that starting both from n- and p-type material, one obtains, after prolonged irradiation, intrinsic material. Whereas, annealing in irradiated germanium is readily observed at room temperature, large annealing effects are observed in irradiated silicon only at elevated temperatures. The behavior of the irradiated material indicates that levels are introduced close to the top and the bottom of the energy gap.

31. Fan, H. Y. and Lark-Horovitz, K.

IRRADIATION EFFECTS IN SEMICONDUCTORS.

Purdue University Department of Physics.

June 57.

Semiconductors are a class of material, the electronic properties of which are most sensitive to imperfections of the crystal lattice. This is evidenced by the controlling effect of small amounts of chemical impurities which are one type of lattice imperfections. Irradiation by high energy particles can produce therefore radical changes in the properties of a semiconductor by creating defects in the crystal lattice. These defects may introduce new energy levels. Thus the electrical conductivity of a semiconductor may change by orders of magnitude as a result of irradiation, whereas in metals the changes are measured in percent. Many other properties are likewise sensitive to irradiation, making irradiation effects in semiconductors of interest.

32. Johnson, W. E. and Lark-Horovitz, K.

NEUTRON IRRADIATION OF SEMI-CONDUCTORS.

Fairchild Engine and Airplane Corporation.

Nepa Division Oak Ridge, Tennessee. 20 Oct 49.

High Resistance P Type Germanium Conductance Versus Time of Irradiation N Type Germanium Sample; Conductance 1 R Versus Time of Irradiation Resistance of Low Resistivity N Type Silicon Plate Versus Time of Irradiation Resistance of High Resistivity P Type Silicon Plate Versus Time of Irradiation P Type Silicon O Versus 1/T After Neutron Bombardment Resistance of IN47 Rectifier When The Resistance Is Determined by Spreading Resistance

Forward Resistance of IN38 Crystal Rectifier Versus Time of Irradiation
Forward & Back Resistance of IN23 Silicon Crystal Rectifier Versus Time of
Irradiation

33. Lord, R. G.

Far infrared transmission of silicon and germanium. PHYSICAL REVIEW v. 85, p. 140-141, Jan-Mar 1952.

34. Lusk, T. E.

Infrared-transmission materials would be unaffected by radiation in space. ELECTRONIC DESIGN v. 8. p. 74, 26 Oct 1960.

Investigation of the transmission properties of 15 infrared optical materials exposed to three levels of gamma radiation - 0.975 x  $10^4$ , 1.58 x  $10^6$ , and 1 x  $10^8$  roentgens. Results are presented in tabular form.....

Philipp, 'H. R. and Taft, E. A.

Optical constants of germanium and silicon
in the region 1 to 10ev. BULLETIN OF THE

AMERICAN PHYSICAL SOCIETY, Series II, v. 4,
p. 27(A), Jan 1959.

Measurements of the reflectance of single-crystal germanium and silicon in the range 0.6 to 11.3ev were reported. The Kramers-Kronig relation was applied to this spectrum and the real and imaginary parts of the complex index of refraction, n and k, were determined. The results for germanium agree with the recent measurements of Archer and extend his values beyond 3.5ev. Pronounced structure is observed in both germanium and silicon in the vicinity of 4.5ev. The extinction coefficient rises to a peak near this energy. At  $80^{\circ}$ K, the peak sharpens and shifts toward higher energy. The strength of this absorption should be useful in testing and refining band structure calculations. Based on the Frohlick-Pelyer criterion, which is applicable to

non-metals, no evidence for plasma oscillations was found. Discrete energy losses have been observed at higher energy, and it would be of considerable interest to extend the present data to that range.

36.

Purdue Research Foundation, Lafayette, Ind.

Rept. no. PRF 1258. Final rept. 1 July 56-

30 Sep 60, 47p. (Contract DA 36-039-sc-71131).

ASTIA AD-251 056.

Contents:
Not carrier studies
Surface studies
Impurity conduction
Optical properties
Magnetic properties
Specific heat
Thermal conductivity and thermoelectric power
Irradiation effects
Crystal growth and structure investigation
Theory

37.

Purdue University Department of Physics SEMICONDUCTOR RESEARCH FIFTH QUARTERIX REPORT 1 July 57-30 Sep 57. (Contract DA 36-039-sc-71131).

Preparation of Materials

Preparation and Analysis of Bulk Semiconductors, Roth, L.

Growing of Silicon Single Crystals. Klose, P.

Theoretical Problems

Level Density Functions in Disordered One-Dimensional Crystals. James, H. M. Optical Properties

The Absorption of Germanium and Tellurium at Long Wavelength. Fisher, P.

and Fan, H. Y.

Infrared Absorption in Tellurium. Caldwell, R. S. and Fan H. Y.

Low Temperature Problems

Apparatus for Measuring Specific Heat down to 0.4°K Using A Bath of He<sup>3</sup>. Seidel, G. and Keeson, P. H.

Electrical Measurements of Silicon at Low Temperature. Ray, R. K., Longo, T. and Lark-Horovitz, K.

Paramagnetic Resonance

Paramagnetic Resonance in Irradiated Silicon, Nisenoff, M. and Fan, H. Y. Electrical Properties.

Electrical Properties of High Resistivity Silicon. Wang, E. Y. and Longo, T. A.

Surface Photoconductive Effects. Cunningham, R. W. and Bray, R. Bombardment Experiments

Alpha-Particle Irradiation of Ge at 4.20K. Gobeli, G. W. and Lark-Horovitz, K.

Changes in Mobility of Charged Carriers Produced by Deuteron Irradiation of Silicon. Longo, T. A.

38. Purdue Research Foundation

SEMICONDUCTOR RESEARCH. QPR no. 6, Dec 52.

(DA-039-sc15339). ASTIA AD 3377.

The infrared spectrum exhibits a group of absorption bands which have been present in all samples examined. In silicon which showed similar absorption bands, nucleon bembardment failed to change the absorption bands in either magnitude or position. The absorption in Ge were taken using samples from several different melts of high purity material. In all cases the results were the same as given in QPR no. 3. In samples of resistivity from 40-0.3 ohm-cm the lattice component is unaffected.

39. Purdue University Department of Physics
SEMICONDUCTOR RESEARCH SIXTH QUARTERIN
REPORT. 1 Oct 57-31 Dec 57. (Contract

DA 36-039-80-71131).

Freparation and Analysis of Materials

Preparation of Germanium and Compound Single Crystals. Roth, L. Growing of Silicon Single Crystals. Klose, P. H. and van der Aa, J. W. D. Double Crystal X-ray Spectrometer. Yearian, H. J.

Optical Properties

Effect of Irradiation on Infrared Absorption in Silicon. Ramdas, A. K. and Fan, H. Y.

Low Temperature Problems

The Specific Heat of Zinc in The Normal and Superconducting States. Keesom, P. H. and Seidel, G.

Magnetic Susceptibility. Gerritsen, A. N. and Damon, D. H.
Paramagnetic Resonance In Irradiated Silicon. Nisenoff, M. and Fan, H. Y.
Low Temperature Specific Heat of Molybdenum Alloys. Bryant, C. A. and
Keesom, P. H.

Transport Properties of Germanium at Low Temperatures. Goff, J. F. and Pearlman, N.

Electrical Properties of Silicon at Low Temperature. Ray, R. K., Longo, T. A. and Lark-Horovitz, K.

Annealing of Germanium Following Alpha-Particle Irradiation at 4.20K. Gobeli, G. W., Fan, H. Y. and Lark-Horovitz, K.

Surface Properties

Surface Sensitive Conductance of High Resistivity Silicon. Wang, E. Y. and Longo, T. A.

Hall Effect Measurement at High Electric Field. Brown, D. and Bray, R. Spectral Dependence of Surface Photoconductivity in Ge. Cunningham, R. W. and Bray, R.

40.

Purdue Research Foundation

SEMICONDUCTOR RESEARCH. QR no. 9, Sep 53.

(Contract DA-36-039-sc 63222). ASTIA

AD=43 314.

A summary of test results for this period can be found in the Summary Report dated 1 July 51-1 July 54: SUMMARY OF SEMICONDUCTOR RESEARCH.

41.

Purdue University Department of Physics

SEMICONDUCTOR RESEARCH TENTH QUARTERLY

REPORT. 1 Nov 58-31 Jan 59. (Contract

DA 36-039-sc-71131).

Thermal Capture of Electrons by Vacancies. Paranjape, B. V.
Preparation of Semiconductor Crystals. Roth, L.
Photoconductivity and Sweepout for Case of Majority Carrier Trapping. Bray, R.
Bulk Conductivity at High Electric Field. Brown, D. and Bray, R.
Impurity Conduction in Silicon. Ray, R. K. and Fan, H. Y.
Transport Properties of Germanium at Low Temperatures. Goff, J. F. and
Pearlman, N.
Impurity Photoconductivity in Germanium. McConville, G. T. and Fan, H. Y.
Infrared Absorption in Irradiated Silicon. Ramdas, A. K. and Fan, H. Y.

Impurity Photoconductivity in Germanium. McConville, G. T. and Fan, N. I.
Infrared Absorption in Irradiated Silicon. Ramdas, A. K. and Fan, H. Y.
Paramagnetic Resonance in Neutron-Irradiated Silicon. Nisenoff, M. and Fan,

H. Y. with Appendix by Wun Jung

Magnetic Susceptibility. Damon, D. H. and Gerritsen, A. N. Anomaly in the Specific Heat of Rhenium. Bryant, C. A. and Keesom, P. H.

42.

Purdue University Dept. of Physics

SEMICONDUCTOR RESEARCH. Final Report.

1 July 54-30 June 56. (Contract DA

36-039-8063222).

Infrared absorption in n-type Germanium.

The investigation was carried out at different temperatures and from 5-30 microns. The results indicate that at high temperatures the lattice scattering becomes important for the absorption.

Infrared absorption in n-type silicon.

The magnitude of the absorption increases with the conductivity of the sample. Measurements have also been made at liquid nitrogen and liquid helium temperatures. The band remains the same. The results suggest that the absorption band corresponds to excitation of conduction electrons or electrons on donor impurities to higher energy bands.

43.

Purdue University. Dept. of Physics.

SUMMARY OF SEMICONDUCTOR RESEARCH. 1 July

51-1 July 54. (Contract DA-36-039-sc 15339).

Infrared lattice absorption bands in germanium, silicon.
Infrared absorption bands characteristics of the crystal lattice were investigated for germanium and silicon. The absorption was found to be insensitive to lattice imperfections due to impurities (~10<sup>18</sup>cm<sup>-3</sup>) and due to disorders (~10<sup>18</sup>cm<sup>-3</sup>) produced by nucleon irradiation.

44.

Raytheon Manufacturing Company Research

Division, Waltham, Massachusetts. STUDY

OF SEMICONDUCTOR MATERIALS AND DEVICES.

Final Report. Index No. Ne-110458-Subtask

4. 1 Oct 55-30 June 57. (Contract

NObsr-72537)

Includes detailed discussion of work done during the Seventh Quarter, 1 Apr 57-30 June 57.

DETAIL FACTUAL DATA

The Behavior of Oxygen in Plastically Deformed Silicon

Optical Studies on Silicon in the Infrared

X-Ray Measurements of Crystal Perfection in Silicon

Dislocations in Silicon

Lifetime Measurements and Apparatus

Semiconductor Surface Studies

Positive Ion Bombardment of Germanium and Silicon

Space-Charge Widening as a Function of Collector Blas for Diffused Junction

Error in the Measurement and Calculation of Surface Concentration

Diffusion Studies

Silicon High-Frequency Transistors

Silicon Power Transistors

High-Frequency Semiconductor Devices Utilizing the Injection of Carriers into Space-Charge Regions

45.

Shaw, C. C. and Krogstad, R. S.

NUCLEAR RADIATION EFFECTS ON INFRARED

MATERIALS AND COMPONENTS. PART I. EFFECTS

OF GAMMA RADIATION ON INFRARED TRANSMITTING

MATERIALS AND FILTERS. Electronics

Division Research and Development Branch,

Lockheed Missile Systems Division, Palo Alto,

California. 16 June 58, 20p.

Results of a survey of cobalt-60 gamma radiation effects on the optical properties of a number of infrared transmitting materials and filters are presented.

This study shows that, with few exceptions, the materials and filters are insensitive to gamma radiation at a dosage level of 107 roentgens or more.

46.

Shilliday, T. S.

Optical effects in semiconductors.

BATTELLE TECHNICAL REVIEW v. 8, n. 9,

p. 7-13, Sep 1959.

The author centers his discussion on the infrared region of the electromagnetic spectrum. He describes the effects of radiation upon semiconductor devices,

discusses the devices of interest in the area, and points out how the phenomena of radiation are used in solid-state research and in technology.

47.

Signal Corps Engineering Laboratories,

Fort Monmouth, N. J. A SURVEY OF THE

NUCLEAR RADIATION EFFECTS ON SEMICONDUCTOR

MATERIALS AND DEVICES. Technical Memorandum

NR. M-1902. 1 June 57.

This report discusses some of the presently available information on the effects of radiation (primarily neutron and gamma radiation) on semiconductor materials and devices. Discrepancies in some of the results obtained are indicated, and suggestions are made for further experimentation.

48.

Spitzer, W. G.

INFRARED PROPERTIES OF SEMICONDUCTORS.

Department of Physics, Purdue University.

June 57. ASTIA AD-142 613.

Abstract

Introduction

Experimental Equipment and Procedures

Monochromators

Absorption Cells

Sample Preparation

Sample Thickness Determination

Technique of Measurement

Intrinsic Absorption Edge in Germanium and Silicon

Spectral Shape

Introduction

Experimental Measurements

Discussion

Germanium

Silicon

Pressure Dependence

Introduction

Experimental Method

Results and Discussion

Germanium

Silicon

Effect of Impurities

```
Carrier Absorption in N-Type Germanium
   Introduction
   Experimental Measurements
   Discussion of Results
Absorption in Silicon
   P-Type Silicon
   N-Type Silicon
   Plastically Pure Silicon
Absorption and Reflectivity of Indium Antimonide
   Introduction
   Experimental Measurements
   Discussion of Results
      From 5 to 35 micron
      From 35 to 90 micron
      Beyond 90 Micron
Determination of Optical Constants and Carrier Eff ctive Mass of Semicond ctors
   Introduction
   Theory
      Electric Susceptibility, Conductivity, and Effective Mass of Free Carriers
      Electric Susceptibility Due to Interband Transitions
   Experimental Method
   Results and Discussion
      N-Type Germanium
      P-Type Germanium
      N-Type and P-Type Silicon
      P-Type Indium Antimonide
      N-Type Indium Antimonide
      N-Type Indium Arsenide
Bibliography
Appendix
Vita
  Figure Legends
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49.

Steel, H. and Owen, D.

EFFECTS OF RADIATION ON LEAD SULFIDE

INFRARED DETECTORS AND INFRARED OPTICS.

Boeing Airplane Company, Seattle Division.

Paper Presented at the 2nd Semi-Annual Weapons

System Radiation Effect Symposium at Battelle

Memorial Institute. 22-23 Oct 57.

A dose of  $2x10^4$ r is sufficient to cause serious changes in the visible part of the spectrum. There is however, lack of change in the IR. It has been determined that 10 nvt doses were necessary to produce 5 to 10% changes in transmission in silicon and germanium.

#### SILICA See Also Citation no. 82.

50. Monk, G. S.

THE COLORATION OF OPTICAL MATERIALS BY

HIGH ENERGY RADIATIONS. University of

Chicago, Argonne National Laboratory. ANL-

4536, 31 July 50.

The coloration by irradiation of fluoride, calcite, and polaroid crystals and of silica, lead, and other optical glasses are discussed.

51. Weeks, R. A. and Nelson, C. M.

Trapped electrons in irradiated quartz and

silica: II. Electron spin resonance.

JOURNAL OF THE AMERICAN CERAMIC SOCIETY

v. 43, n. 8, p. 399-404, 1 Aug 60.

The correlation of two paramagnetic defects, observed by the electron spin resonance (ESR) technique, with two optical absorption bands produced by & -ray or neutron irradiation is indicated. The peaks of the two absorption bands fall at ~2100 a.u. of 2300 a.u.

#### FUSED SILICA

52. Arnold, G. W. and Compton, D.

Radiation effects in silica at low temperature

PHYSICAL REVIEW v. 116, n. 4, Ser. 2,

p. 802-811, 15 Nov 1959.

Optical absorption bands induced in fused silica and crystalline  $\alpha$ - quartz of low impurity content at  $77^{\circ}$ K by fast electrons of X-rays bleach slowly at room temperature. The presence of OH- ions in fused silica inhibits the formation of such radiation-induced absorption.

53. Gale, A. J. and Bickford, F. A.

Radiation-resistant fused silica. NUCLEONICS

v. 11, n. 8, p. 48, Aug 1953.

Tests carried cut at High Voltage Engineering Corporation on insulating materials for accelerator tubes include high-energy electron bombardment of materials, mainly glasses, proposed or intended for this use.

54. Levy, Paul W.

Reactor and gamma ray induced coloring in

Corning fused silica. BULLETIN OF THE AMERICAN

PHYSICAL SOCIETY v. 30, n. 2, p. 18, 17 Mar 1955.

The coloring induced by gamma ray and reactor radiations in fused silicas from various manufacturers and in Brazillian crystalline quartz has been studied by optical transmission measurements in the region 2000 A to 1 micron.

55. Levy, P. W.

Reactor and gamma ray induced coloring in Corning fused silica (Abstract of meeting) PHYSICAL REVIEW v. 98, n. 5, p. 1541, 1 June 1955.

The coloring induced by gamma ray and reactor radiations in fused silica from various manufactures and in Brazilian crystalline quartz has been studied by optical transmission measurements in the region 2000 A to 1 micron. Corning purified fused silica shown practically no gamma ray coloring while reactor irradiaton produces a strong band at 2180 A, a weak band at 2420 A, and very slight coloring in the visible.

56. Levy, P. W.

Reactor and gamma-ray induced coloring of Corning fused silica. J. PHYS. CHEM .SOLIDS v. 13. n. 3-4, p. 287-95. June 1960.

When crystalling quartz, fused silica and many other substances are subject to reactor or gamma-ray radiations the samples develop optical absorption bands in the region 0.2-1 µ. Usually, there are many broad bands which develop and resolution of the observed spectrum into the component bands is difficult. Corning fused silica is coloured less than all of the materials studied and the observed spectrum can be resolved into its components by properly utilizing the variations in the relative absorption of the different bands created by changing the irradiation conditions. The most intense band is mear 5.75 eV (218 mu) and there is a smaller one at 5.05 eV (242 mu) when the sample is irradiated in the reactor at 70°C. The 5.05 eV band has, relative to the 5.75 eV band, low intensity when the reactor irradiation is at 170°C but is much stronger when the irradiation is at liquid nitrogen temperature. Also, the peak of the 5.74 eV band shifts slightly with irradiation temperature. The band at 5.05 eV, which can be separated from the other bands without assuming a shape for it, is well fitted by a Gaussian curve and it is assumed that all other bands are similarly shaped. When a sample originally coloured in the reactor is subsequently subjected to gamma-rays, additional absorption bands appear and their intensity is proportional to both the reactor and gamma-ray exposures. In all, new bands at approximately 5.5, 4.5, 2.0 and ~6.1 eV (223, 278, 625 and 200 mm, respectively) are present with indications of four others.

Nelson, C. M. and Crawford, J. H., Jr. Optical absorption in irradiated quartz and fused silica. J. PHYS. CHEM. SOLIDS v. 13, n. 3-4, p. 296-305, June 1960.

Optical absorption studies over the range from 1850 to 26000 A have been performed on crystalline quartz and fused silica after exposure to both fast neutrons in the reactor and  ${\rm Co}^{60}$  Y-rays. The prominent band near 2150 A is produced by  $\gamma$ -rays and neutrons in fused silica but fast neutron exposures in excess of 5 x  $10^{18}$  cm<sup>-2</sup> are necessary to cause appreciable development of the corresponding band in crystalline quartz. With shorter neutron exposures and \( \gamma\)-rays the absorption in quartz crystals is characterized by the broad visible bands and continuous absorption which rises almost linearly toward shorter wavelength below 3000 A. This continuous absorption probably corresponds to the long wavelength tail of one of the bands observed by Mitchell and Paige in the vacuum ultraviolet region. These results do not seem to agree with their interpretation of the nature of the 2200 A band in quartz. On annealing neutron-irradiated quartz (5 x 10<sup>18</sup> neutrons/cm<sup>2</sup>) at 250°C a new band at 2000 A develops. In fused silica, besides the 2150 A band, the shoulder at 2400 A (resolved by appropriate optical bleaching as a band at 2570 A) which has previously been reported, was observed with both Y-rays and fast neutrons. The rates of coloration of quartz and fused silica as well as studies of optical and thermal bleaching are discussed. In the fused materials a mechanism of photolytic coloration which appears to be consistent with observations provides a suitable model which is based on the rupture of Si-O bonds with the formation of free radicals as the principal product. These and other possible defects are discussed in the light of magnetic susceptibility and electron spin resonance studies.

Nelson, C. M. and Crawford, J. H., Jr.

OPTICAL ABSORPTION IN IRRADIATED QUARTZ

AND FUSED SILICA. Proceedings of Conference
on Defect Structures of Quartz and Glassy

Stlicas, Book, John Wiley and Sons.

The work described here was initiated in an attempt to learn more about the chemical and electronic effects that must accompany the loss of structure in quartz after prolonged neutron irradiation. To this end, measurements of optical absorption spectra, paramagnetic resonance, and magnetic susceptibility on various forms of silica as a function of neutron and gammaray exposure were udertaken. The present paper may be considered a progress report on the optical portion of this work. Included here is a summary of

the rate of coloration with both gamma rays and neutrons, the optical and thermal bleaching behavior and the effect of previous bombardment on the susceptibility of fused silica to gamma-ray coloration.

Equipment and methods for producing radiophotoluminescent glass at a rate of 5,000 pieces of specified dimensions per day were developed. The radiophotoluminescent glass is the sensitive element in the DT-60 ( ) PD Radiac Detector as specified in Interim Military Specification MIL-D-16153C (SHIPS). This element is capable of indicating an accumulative dosage of X- and gamma radiation over a range from 0 to 600 roentgens. The glass is made in this program, as represented by melt number E-9880, successfully meets the requirements of specification MIL-D-16153C (SHIPS) to the limits that it was tested.

## VITREOUS SILICA

59.

Simon, I.

Structure of neutron-irradiated quartz and vitreous silica. JOURNAL OF THE AMERICAN

CERAMIC SOCIETY v. 40, n. 6, p. 150-153, June 1957.

The structure of quartz and vitreous silica disordered by heavy irradiation (10<sup>20</sup> neutrons/cm<sup>2</sup>) of fast neutrons was studied by means of X-ray diffraction and infrared spectroscopy. The results indicate that the structure of the disordered amorphous form of silica resembles closely that of vitreous silica. It is suggested that the disordered form of silica results from thermal effects (thermal spikes) accompanying the passage of knocked-on atoms through the solid.

60.

Primak, W.

Fast-neutron-induced changes in quartz and vitreous silica. PHYSICAL REVIEW v. 110, n. 6, p. 1240-1254, 15 June 1958.

The course of the changes produced by exposure in nuclear reactors and the subsequent alteration of the radiation-induced property changes caused by heating were followed by measuring the dilatation, refractive index, rotatory power and birefringence.

61.

Primak, W. and Uphaus, R. A.

Fast neutron induced luminescence in vitreous silica and quartz. JOURNAL OF CHEMICAL PHYSICS

v. 29, n. 4, p. 972-973, Oct 1958.

An attempt to measure light scattering by vitreous silica and quartz which had been exposed in a nuclear reactor proved unsuccessful because of the appearance of a reddish luminescence much more intense than the scattered light. Since no reports of a red luminescence could be found, it was studied further.

## GLASSES See Also Citation no. 51, 55

62. Best, J. V. F.

THE COLOURATION OF SOME OPTICAL GLASSES

BY X- AND GAMMA RADIATION. Atomic Energy

Research Establishment, Harwell, England.

A.E.R.E. C/R 1125. 10 Mar 53. ASTIA AD-11 678.

Samples of optical and other glasses have been subjected to large doses of X-and gamma rays and their resistance to colouration studied. Optical glasses are more prone to colouration than plate glass and the torosilicate compositions used for chemical glassware. Perspex, polystyrene and two European glasses containing small amounts of cerium are very resistant to colouration. A brief summary of recent American experience in this field is appended.

63. Colp, J. L. and Woodall, H. N.

Effects of high neutron and gamma fluxes on

transmission characteristics of some optical

glasses. Paper presented at the 61st Annual

Meeting, SYMPOSTUM ON RADIATION EFFECTS ON

MATERIALS, Boston Massachusetts, 24 June 1958.

vol. 3, ASTM Special Technical Publication .

No. 233, p. 156-163.

Specimens of several types of optical glass most of which were of the radiation-tolerant type were exposed to gamma radiation levels of 10<sup>6</sup>, 10<sup>7</sup>, and 10<sup>9</sup> r in the Sandia kilocurie cobalt-60 facility.

Exposures of  $10^{10}$  r were made at the Materials Testing Reactor gamma facility. Exposures to mixed reactor radiation (gamma and neutron) were also made at the Materials Testing Reactor.

The light transmission properties of the glass specimens were measured before and immediately after exposure to radiation.

64. Ferguson, K. R.

Optical absorption bands induced in gamma irradiated glass (Abstract of meeting)

PHYSICAL REVIEW v. 98, n. 5, p. 1542,

1 June 1955.

The formation of optical absorption bands in glasses exposed to Co-60 gamma rays has been observed.

65. Kazen, D. and Reiffel, L.

RADIATION SENSITIVE GLASSES. Armour Research

Foundation, Chicago, Illinois, Quarterly

Report No. 4. REIC No. 5906. 1 Mar-1 June 53.

(Contract DA 36-039-sc-15528). ASTIA AD-16 150.

Validity of the "ordering" hypothesis has been strengthened by a repetition of the heat treatment and X-ray diffraction studies of K-48 glass. The observation of reversal to the strength of the two X-ray lines in a single piece of glass in a similar manner of that previously observed in two separate samples rules out composition differences as a factor. Irradiation experiments performed with a series of BC-2 glasses indicate that effects similar to those produced by heat treatment can be produced by a reduction in the alkali ion content of barium crown glasses.

66. Kreidl, N. J. and Hensler, J. R.

Gamma radiation insensitive optical glasses.

JOURNAL OF OPTICAL SOCIETY OF AMERICA v. 47,

n. 1, p. 73-75, Jan 1957.

Specially developed optical glasses maintain their transparency under exposures up to  $10^6$  roentgens cobalt-60 gamma radiation, and are usable at 5 x  $10^6$  roentgens. Ordinary optical glass colors at  $10^4$  roentgens or less and is nearly useless at  $10^6$  roentgens.

Kreidl, N. J.

SOME EXPERIMENTS ON THE INTERACTION OF
GLASS WITH HIGH ENERGY RADIATION. Bausch
and Lomb Optical Company. NYO-3779. 1 Dec 53.
(Contract AT-30-1-1312).

A number of experiments dealing with the effects of radiation on optical glass are summarized.

Kreidl, N. J. and Blair, G. E.

IRRADIATION DAMAGE TO GLASS. Bausch and
Lomb Optical Company, Rochester, New York.

NYO-3782. 21 Mar 55. (Contract AT(30-1)-1312). ASTIA AD-64 301.

A system of high-level dosimetry (of high-energy radiation) utilizes the absorption changes in glasses measured on a suitable spectrophotometer. Calibration has been accomplished over the range of  $1.5 \times 10^3$  to  $4.1 \times 10^6$  rep.

Mayer, G. and Gueron, J.

Kinetics of the bleaching of glasses colored by irradiation in the chatillon pile. THE GLASS INDUSTRY v. 34, n. 3, p. 127-131, 158-159, Mar 1953.

Samples of glass and silica were colored by irradiation in the Chatillon pile. The absorption spectra of the irradiated samples were studied between 200 and 2000 mm.

With the aid of the theories on alkali halides, the interpretation of the numerical values obtained for absolute rates of reaction, for experimental activation energies and orders of reaction, was also attempted.

Reiffel, L. and Humphreys, R. F.

RADIATION SENSITIVE GLASSES. Armour Research

Foundation, Chicago, Illinois. Quarterly

Progress Report No. 3. 10 Mar 53. (Contract

DA-36-039-SC-15528). ASTIA AD 9426.

Work during the period of this report has established some of the characteristics of radiation induced color centers, particularly with regard to stability and absorption characteristics. Work on the energy transfer mechanism particularly as a function of temperature is still ambiguous. "High temperature" glasses seem to exhibit more appreciable radiation sensitivity.

71.

Reynolds, R. L.

TRANSPARENCY TO FAR INFRARED. General

Electric Company, Hanford Atomic Products

Operation, Nonmetallic Materials Development

Operation. HW-39536. 15 Oct 55.

Of the six optical materials tested following exposure to 1.5 x  $10^6$  r, arsenic trisulfide glass was shown to transmit infrared to beyond  $10\mu$ , and was essentially unaffected by radiation.

72.

Schulman, J. H., et al.

The effect of high energy radiation on the absorption and luminescence of glasses and crystals (Abstract from meeting). THE

AMERICAN CERAMIC SOCIETY BULLETIN v. 30,

p. 97, 1951.

The behavior of a number of crystalline and glassy systems will be described, and an interpretation of the phenomenon will be proposed for some of the simpler cases.

73. Sun, K. H. and Kreidl, N. J.

DISCOLORATION OF GLASS BY RADIATIONS.

Bausch and Lomb Optical Company, Rochester,

New York. Special Report, NObsr 57016.

May 52. ASTIA AD-120 816.

This report was prepared as a fundamental background for all workers in the title field. It is large in its scope, detailed in its presentation. It included a review based on a large bibliography.

74. Weyl, W. A. and Otley, K. O.

EFFECTS OF RADIATIONS UPON GLASSES. The

Pennsylvania State College, School of Mineral

Industries. First Quarterly Progress Report.

1 July-30 Sep 50. (Contract DA-36+039-sc-132).

ASTIA ATI 89918.

A survey of the existing literature on the effect of nuclear radiation on glasses is given. Silver containing phosphate glasses exhibit a bright orange or pink fluorescence after exposure to X-rays. Various silicate glasses which all contained identical amounts of vanadium and cerium oxide were exposed to solar radiation. A glass composition was thus found which showed a noticeable change of color after only five minutes' exposure to bright sunlight.

QUARTZ
See Also Catations no. 52-57, 59-63, 79, 82

75.

Arnolca, G. W., Jr.

Color centers in synthetic quartz.

THE JOURNAL OF CHEMICAL PHYSICS . v. 22,

n. 7, p. 1959-1260, July 1954. (REIC NO. 5059).

This note is written to report an absorption maximum in X-irradiated synthetic quartz to 2.64 ev (470µ). Samples of synthetic quartz cut perpendicular to the c axis yielded the 2.64 ev maximum when X-irradiated by means of a tungsten tube operated at 45 kv at 15 ma for periods from 24-44 hours.

76.

Arnolæ, G. W., Jr.

Defects in natural and synthetic quartz.

J. PHYS. CHEM. SOLIDS v. 13, n. 3-4, p. 306-20,

June 1\_960.

Thermoluminescence measurements of quartz (both irradiated and naturally coloured) have been made from room temperature to 375°C. Various peaks of the light intensity versus temperature curves have been identified with absorption maxima in the L-9-1000 my spectral region. The method is extremely sensitive to radiation dosse; well-defined peaks being observed for very short X-irradiation times (~1 see). Data are presented on X-irradiated natural quartz, smoky quartz and con X-irradiated synthetic quartz with various additives.

77.

Aruturmian, G. and Renius, O.

THE EFTECTS OF X-RADIATION ON THE OPTICAL

PROPERTIES OF QUARTZ CRYSTALS. Report of

Conference on Effects of Nuclear Radiation

on Materials sponsored by Ordnance

Materials Research Office at Watertown

Arsenal. 1-2 Oct 57, Feb 58.

The object of this paper is to review briefly the changes in the optical properties of polished quartz primarily in the spectral regions where the human eye is not sensitive such as the ultraviolet and near infrared spectral regions.

78. Bechmann, R.

Radiation effects in quartz - A bibliography.

NUCLEONICS v. 16, n. 3, p. 122, Mar 1958.

Radiation causes quartz crystals to display changes in color, thermal and electrical conductivity, index of refraction, and elastic properties. This is a bibliography of literature that describes investigations of these effects. A brief discussion of the effects follows a listing by type.

79. Brown, C. S. and Thomas, L. A.

Response of synthetic quartz to x- ray
irradiation. NATURE v. 169, n. 4288,
p. 35-36, Jan 1952.

It is concluded from the results with irradiated synthetic crystals that the darkening in natural quartz may be associated with the presence of impurities, and that changes occurring during the growth of a crystal often produce corresponding changes in the susceptibility to X-ray darkening in the different parts of the crystal.

80. Frondel, C. and Hurlbut, C. S.

INVESTIGATION INTO EFFECTS OF RADIATION

ON THE PHYSICAL PROPERTIES OF QUARTZ.

Harvard U., Cambridge, Mass. Final rept.

1 July 51 - 30 July 53, 53p. (Contract
DA 36-039-sc-15350). ASTIA AD-20 661.

The work on synthetic and natural quartz involved quantitative chemical analyses and measurements of the physical properties. The latter included density, index of refraction, high-low inversion temperature, fluorescence and triboluminescence, rate of solution and unit cell dimension measurements. The variation in properties and composition of quartz was established; however, a clear-cut detailed correlation between the variation in different samples was not obtained. Extreme cases of variation indicated that the chief variable in quartz was a solid solution of Al for Si in the crystal structure. The irradiation behavior of quartz appeared to be related to the composition.

81.

Klemens, P. G.

Density changes in neutron irradiated quartz. PHILOSOPHICAL MAGAZINE v. 1, n. 10, Ser. 8, p. 938-941, Oct 1956.

Wittels (1953) and Wittels and Sherrill (1954) measured the density changes produced in quartz by fast-neutron irradiation in a pile. The density was found to decrease with increasing dose, and the density change attained a saturation value of about 15 per cent for doses in excess of 2 x 10<sup>20</sup> neutrons/cm<sup>2</sup>. However, the rate of density change did not decrease steadily, but increased at first. A different explanation of this increase in the rate of density change is given here.

82.

Lautout, M.

Photostimulation and coloration of fused quartz irradiated by X- or gamma radiation.

(Photostimulation et coloration du quartz fondu irradie par les rayons X our gamma) JOURNAL

DE CHIMIE PHYSIQUE v. 52, p. 267-271, 1955.

Fused quartz, previously irradiated by X-rays or gamma rays, becomes phosphorescent under the action of visible or ultra violet light.

The exciting light first causes, on the other hand, a change of color of the quartz which appears streaked with dark lines, sometimes appearing as vortexes.

83. Mitchell, E. W. J. and Paige, E. G. S.

The optical effects of radiation induced atomic damage in quartz. PHILOSOPHICAL MAGAZINE v. 1, n. 12, Ser 8, p. 1085-1115,

Dec 1956.

The optical absorption of neutron-irradiated crystalline and fused quartz has been measured in the wavelength range 1450 to 10,000 Å (8.5 to 1.2 electron-volts). Two absorption bands associated with atomic displacements in crystalline quartz have been found -- C at 5.7 electron - volts and E at 7.6 electron - volts. All the damage is annealed by heating at 950 C.

84. Mitchell, E. W. J. and Regden, J. D.

The effects of radiation on the mear infrared absorption spectrum of alpha-quartz.

PHILISOPHICAL MAGAZINE v. 2, n. 20, Ser 8,
p. 941-956, Aug 1957.

The variation of the absorption strength and spectrum in the region of  $3\mu$  has been determined for different specimens of crystalline quartz and for X-ray, electron and pile irradiated crystals.

Mitchell, E. W. J. and Wedepohl, P. T.

The scattering of long wavelength neutrons
by irradiated and unirradiated quartz.

PHILOSOPHICAL MAGAZINE v. 3, Ser 8, p. 12801286, 1958.

Measurements have been made of the transmission of neutrons of wavelengths between 50 and 10A through 10 cm of unirradiated and pile irradiated Brazilian quartz crystals. After irradiation with 6.4 x 1018 fast  $\rm n^{\circ}$  cm  $^{-2}$  additional scattering was found. The measurement is discussed in relation to optical absorption measurements and radiation damage theory.

Wittels, M.

The lattice expansion of quartz due to

fast neutron bombardment. THE PHYSICAL

REVIEW v. 89, n. 3, p. 656-657, 1 Feb 1953.

The progressive anisotropic expansion of a-quartz irradiated with increasing dosages of fast neutron flux has been observed and measured by X-ray and density methods of analysis. Bombardment produced by a neutron source that gave a total integrated neutron flux of  $6.6 \times 10^{-9}$  nvt resulted in a density change of  $(\Delta p/p)x$  100 = 3.5 + 0.1 as measured by the hydrostatic weighting method. The density change measured by the X-ray method gave  $(\Delta p/p) \times 100 = 4.8 \pm 1.5$ . The crystal temperature was approximately 100 C during irradiation.

87.

Wittels, M. C.

STRUCTURAL BEHAVIOUR OF NEUTRON IRRADIATED

QUARTZ. Paper submitted for publication to

Philosophical Magazine.

X-ray and density studies have been made on single crystals of quartz irradiated with fast pile neutrons at approximately 100°C. The structural effects of the irradiation follow at least two processes with doses up to 1.2 x 10²°C neutrons/cm². Crystals irradiated with less than 3 x 10²°C neutrons/cm² expand anisotropically in a manner which is similar to the thermal expansion of unirradiated quartz. In this dosage range, the volume increase is the same. After doses in excess of 3 x 10²°C neutrons/cm² the damaging process is more complex since the volume change indicated by X-ray measurements of the host lattice becomes larger than the bulk volume change determined hydorstatically. In a complementary study, it was found that coesite, a high density crystalline silica remained stable under neutron irradiation dosages which completely disorder quartz. These results indicate that the rate of damage in silica solids is structure dependent.

88.

Wood, D. L.

Infrared absorption of defects in quartz.

J. PHYS. CHEM. SOLIDS v. 13, n. 3-4,

p. 326-36, June 1960.

A number of sharp absorption bands in the infrared spectrum of crystalline  $\alpha$ -quartz between 3200 cm<sup>-1</sup> and 3655 cm<sup>-1</sup> have been assigned to defects in the

lattice. Defects containing water may cause a broad absorption through this region of the spectrum in very imperfect parts of certain crystals, but it does not contribute to the sharp bands observed. One band at 3581 cm<sup>-1</sup> is probably due to the OH stretching vibration of a proton defect but the other peaks are not. Treatments of the crystal involving X-irradiation, heat bleaching, annealing or heating in an electric field strongly affect the intensities of the bands observed, and it is concluded that the bands originate in colour centres with an electronic origin. Probably more than one type of centre is involved. No correlation with the visible colour centres of smoky quartz or amethyst is observed. Interstitial foreign atoms play an important role in the formation of the infrared centres but vacancies or interstitial Si or O atoms are probably not involved. Although much information relating to the infrared colour centres is available the full details of their structure are not yet known.

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<sup>45</sup> YqoO əldslisvA taəE

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